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DETERMINATION OF MERCURY BY COLD-VAPOR ATOMIC ABSORPTION (CVAA) (EPA/SW-846 Methods 7000A/7470A/7471A)

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* These sections affected by Revision 3.0

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on Environmental Protection Agency (EPA) SW-846 Methods 7000A, 7470A and 7471A and those requirements set forth in the latest approved version of the National Environmental Laboratory Accreditation Committee (NELAC) Quality Systems section. Mercury (Hg) in solution may be readily determined by cold-vapor atomic absorption (CVAA) spectroscopy. The method is simple, rapid, and applicable to drinking, surface, and saline waters. Tissue samples, domestic and industrial wastes, water, waste water, extracts, soils, sludges, sediments, and other solid wastes require digestion prior to analysis. The typical reporting limit (RL) is 0.200 micrograms per liter ($\mu\text{g/L}$) for water samples and 0.0400 milligrams per kilogram (mg/kg) for solid samples without correcting for moisture.

This method may not be changed without the expressed approval of the Inorganic Group Leader, the Analytical Section Leader and the Quality Assurance Officer (QAO). Only those versions issued through the REAC document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

2.0 METHOD SUMMARY

Prior to analysis, aqueous and solid samples must be prepared according to the procedures outlined in Section 7.3. The prepared sample with Hg present enters the automated mercury system and is mixed with a reducing agent (stannous chloride) to form elemental Hg vapor. The mixture flows into a liquid-gas mixer where an inert gas is introduced to carry the Hg vapor. The gaseous-liquid mixture enters the liquid-gas separator where the liquid is drained away and the Hg-containing gas continues to the drying tube that dehumidifies the gaseous mixture. The dry vapor then enters the dual beam optical cell that has been optimized for fast response time and high sensitivity. A Hg lamp controlled by the error signal of the reference beam delivers a stable source of emission at 253.7 nanometers (nm). Absorbance by the Hg cold vapor is measured using a solid state detector with a wide dynamic range.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Sample holding times, suggested collection volume, preservative, and type of containers are as follows:

<u>Mercury Measurement</u>	<u>Volume/Weight Required</u>	<u>Type of Containers</u>	<u>Preservative</u>	<u>Holding Time</u>
Total	1000 mL	P, G	HNO ₃ to pH <2	28 days
Dissolved	1000 mL	P, G	Filter HNO ₃ to pH <2	28 days
Solids	200g	G	none/4 °C	28 days
Tissues	10 g	G	none/<10 °C	28 days (Once thawed)

P - plastic, G - glass, g - grams, mL - milliliters, °C - degrees Celsius, HNO₃ - nitric acid, < - less than

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4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Although the method minimizes dilution and sample matrix effects, the technique is not completely interference free. Inorganic and organic Hg compounds may cause a positive interference. Interferences have been reported for samples containing sulfide, chloride, and copper. Organic compounds which have broad band ultraviolet (UV) absorbance (around 253.7 nm) will interfere. Certain volatile organic materials that absorb at this wavelength may also cause interference. Potassium permanganate is added to eliminate possible interference from sulfide. Samples high in chlorides require additional permanganate because chlorine also absorbs at 253.7 nm. The concentration levels for interfering elements/compounds are difficult to define, therefore, quality control (QC) procedures (section 9.0) must be strictly followed.

Cross-contamination and contamination of the sample can be major sources of error due to the sensitivities achieved with the CVAA spectroscopy technique. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as specified in Section 7.1.

5.0 EQUIPMENT/APPARATUS

- Automated Hg Analyzer, consisting of a PS200II analyzer, personal computer, video monitor and printer. Alternatively, a single- or dual-channel, single- or double-beam atomic absorption instrument having a grating monochromator, Hg hollow cathode lamp, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip-chart recorder or computer, printer, autosampler, and Hg cold-vapor generation system may be used.
- Water Bath or Hot Plate, commercially available, capable of maintaining water temperature at 90 +/- 5 °C
- Syringes, commercially available, Henke SASS plastic or equivalent. Alternatively, disposable glass or plastic funnels may be used.
- Filters, 0.45 microns (µm), Corning SCFA or equivalent. Alternatively, glass wool may be used.
- Autopipettors, 50 to 1000 microliters (µL) and 100 to 5000 µL, calibrated quarterly
- Volumetric flasks, Class A, 100 milliliters (mL)
- Glass dispensers, 2-liter (L), 1-L, 1-gallon, checked quarterly for accuracy
- Bottles, 30-mL or 60 mL, Nalgene or equivalent
- Polypropylene round-bottom tubes, 14-mL, for samples
- Graduated cylinder, Class A, 100 mL

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- Polypropylene round-bottom tubes, 50-mL, for standards
- BOD bottles, 300 mL, with ground glass stoppers for digestion of samples and standards
- Bottles, Nalgene or equivalent, miscellaneous volumes for reagent storage
- Tube rack
- Balance, capable of weighing samples to 0.01 grams (g), calibrated annually by an outside vendor

6.0 REAGENTS

- Deionized (DI) water, Type I Water (American Society for Testing and Materials [ASTM] D1193), for the preparation of all reagents and calibration standards and as dilution water
- Nitric Acid (HNO_3), concentrated, trace metal grade
- HNO_3 , 2% - Dilute 20 mL of concentrated trace metal grade HNO_3 to 1-L with DI water in a Class A volumetric flask.
- Hydrochloric Acid (HCl), concentrated, trace metal grade
- Sulfuric Acid (H_2SO_4), concentrated, trace metal grade
- Aqua Regia - Add 3 parts of concentrated HCl to 1 part of concentrated HNO_3 .
- Stock Hg Calibration Standard, commercially available, accompanied by a certificate of analysis, or prepare a 1000 micrograms per milliliter ($\mu\text{g/mL}$) stock standard solution from high purity mercuric oxide (HgO) using DI water and redistilled HCl . Dissolve 1.0798 grams (g) of dry HgO in 50 mL of 1:1 HCl , then dilute to 1-L with DI water.
- Intermediate Hg Calibration Standard, 100 $\mu\text{g/L}$ - Add 0.1 mL of the stock calibration standard to a 100-mL Class A volumetric flask and dilute to 100 mL with 2% HNO_3 . Alternatively, make serial dilutions using 2% HNO_3 to achieve a final concentration of 100 $\mu\text{g/L}$.
- Intermediate Hg Calibration Standard, 10 $\mu\text{g/L}$ - Add 10 mL of the 100 $\mu\text{g/L}$ intermediate Hg calibration standard to a 100-mL Class A volumetric flask and dilute to 100 mL with 2% HNO_3 .
- Laboratory Control Sample (LCS), commercially available, accompanied by a certificate of analysis

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- Stock Hg Initial Calibration Verification/Continuing Calibration Verification Standard (ICV/CCV), 1000 µg/mL, commercially available, accompanied by a certificate of analysis - must be a source different than that used for calibration
- Intermediate Hg ICV/CCV Standard, 100 µg/L - Add 0.1 mL of the stock Hg ICV/CCV to a 100-mL Class A volumetric flask and dilute to 100 mL with 2% HNO₃. Alternatively, make serial dilutions using 2% HNO₃ to achieve a final concentration of 100 µg/L.
- Working Hg ICV/CCV Standard, 2 µg/L - Pipet 2 mL of the 100 µg/L intermediate Hg ICV/CCV standard into a 100-mL Class A volumetric flask and dilute with DI water.
- Stannous Chloride Solution - Dissolve 20g reagent grade stannous chloride (SnCl₂) in 40 mL of concentrated HCl. Slowly add this solution to 100 mL DI water. Dilute to final volume (200 mL) with DI water and mix well. Prepare fresh daily or each time calibration standards are prepared.
- Potassium Permanganate Solution, 5 percent (%) weight/volume (w/v) - Dissolve 100g of Hg-free reagent grade potassium permanganate (KMnO₄) in 2000 mL DI water.
- Sodium Chloride-Hydroxylamine Hydrochloride Solution - Dissolve 12.0 g of reagent grade hydroxylamine hydrochloride [(NH₂OH)₂•HCl] and 12.0 g of sodium chloride (NaCl) in DI water and dilute to 100 mL.
- Potassium Persulfate Solution, 5% w/v - Dissolve 5 g reagent grade potassium persulfate (K₂S₂O₈) in 100 mL DI water.

NOTE: Premixed certified standards will be stored according to the manufacturer's documented storage requirements. These standards may be kept in storage up to the manufacturer's stated expiration date. Once dilutions are made, the standards will be stored for a period not to exceed six months or the manufacturer's expiration date, whichever is less.

NOTE: All calibration standards and spiking solutions will be prepared and documented in accordance with REAC SOP #1012, *Preparation of Standard Solutions and Reagents*.

NOTE: Stock concentrations may vary depending on the vendor; thus, the concentration of intermediate standards may also vary. Preparation instructions may differ based on the volumes prepared and the concentrations of standards available. Any reagent may be prepared at any volume as long as the weight/volume ratios are maintained.

7.0 PROCEDURES

Preliminary treatment of waste water, ground water, extracts/leachates, and industrial waste is always necessary

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because of the complexity and variability of sample matrices. Tissue samples, solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary depending on the nature of the sample being analyzed. NOTE: All chemicals must be added in the order indicated in section 7.1.

7.1 Glassware Preparation

All glassware, polypropylene, or Teflon® containers, including sample bottles, volumetric flasks and graduated cylinders, should be washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and DI water. If it can be documented through an active analytical quality control (QC) program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

7.2 Total Solids

Total solids analysis will be conducted in accordance with REAC SOP #1843, *Determination of Total Solids in Solid Samples*.

A total solids analysis must be conducted for soil, sediment, solid, semisolid and tissue samples to report data on a dry weight basis. Refer to REAC SOP #1843, *Determination of Total Solids*.

7.3 Sample Preparation

7.3.1 Soil, Sediment, Solid, and Semisolid Samples

Weigh 0.2 to 0.8 g (wet) portions of untreated sample and place in the bottom of a BOD bottle. Add 5mL of DI water and 5mL of aqua regia. Heat two minutes in a water bath at 90 +/- 5 °C. Cool; then add 50mL DI water and 15 mL potassium permanganate solution to each bottle. Mix thoroughly and place in the water bath for 30 minutes at 90 ± 5°C.

NOTE: Record the digestion time in and the digestion time out on the digestion log.

Cool and add 6 mL sodium chloride-hydroxylamine hydrochloride solution to reduce excess permanganate. Transfer the sample through a disposable polypropylene funnel into a 100-mL Class A volumetric flask and dilute to final volume with DI water. Filter an aliquot of the sample using a plastic syringe and a 0.45 µm filter into a Nalgene bottle (or equivalent) prior to analysis.

7.3.2 Aqueous Samples

Check the pH of the water sample to ensure that the pH is less than (<)2. If the pH is not <2, adjust the sample pH to <2 by adding additional HNO₃, not to exceed 1% of the total volume of sample. Transfer 100 mL or an aliquot of the sample diluted to 100 mL to a 300-mL BOD

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bottle. Add 5 mL H_2SO_4 and then 2.5 mL HNO_3 , mixing after each addition. Add 15 mL potassium permanganate solution to each sample bottle. Shake and add additional permanganate as necessary, until purple color persists for at least 15 minutes. Sewage samples may require additional permanganate solution. Add the same volume of permanganate solution to all standards, blanks and samples. Add 8 mL of potassium persulfate to each bottle and heat for 2 hours in a water bath at $90 \pm 5^\circ\text{C}$.

NOTE: Record the digestion time in and the digestion time out on the digestion log.

Cool, then add 6 mL of sodium chloride-hydroxylamine hydrochloride to reduce excess permanganate. If necessary, filter an aliquot of the sample using a plastic syringe and a $0.45\ \mu\text{m}$ filter into a Nalgene bottle (or equivalent) prior to analysis.

7.3.3 Tissue Samples

Carefully weigh out a 0.2 to 0.8 g (wet) portion of each sample into a BOD bottle. Do not allow any of the sample to adhere to the sides of the bottle. Add 5 mL aqua regia and 5 mL DI water to each bottle and place in a water bath at $90 \pm 5^\circ\text{C}$ until the tissue is completely dissolved (if necessary, do not dilute aqua regia with 5 mL of DI water in this first step). Cool, then add 50 mL DI water and 15 mL potassium permanganate solution to each sample bottle. Add additional (10 mL maximum) permanganate solution, as necessary, to maintain oxidizing conditions. Add the same volume of permanganate solution to all standards, blanks and samples. Heat in a water bath at $90 \pm 5^\circ\text{C}$ for approximately 30 minutes to solubilize the tissue samples.

NOTE: Record the digestion time in and the digestion time out on the digestion log.

Cool the samples and reduce with 6 mL of sodium chloride-hydroxylamine hydrochloride. Transfer the sample through a disposable polypropylene funnel with glass wool into a 100-mL Class A volumetric flask and dilute to final volume with DI water. Filter an aliquot of the sample using a plastic syringe and a $0.45\ \mu\text{m}$ filter into a Nalgene bottle (or equivalent) prior to analysis.

7.4 Preparation of Working Calibration Standards

For those instruments which do not read out directly in concentration, a calibration curve will be prepared to cover the appropriate concentration range. For best results, working standards should be prepared fresh each time a batch of samples is analyzed. A blank and a minimum of five working standards will be used to calibrate the Hg instrument.

From the 10 $\mu\text{g/L}$ intermediate calibration standard, prepare the following working standards:

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Standard Concentration, $\mu\text{g/L}$	100 $\mu\text{g/L}$ Intermediate Cal Standard, mL	Final Volume
0.2	2	100
0.5	5	100
1.0	10	100

From the 100 $\mu\text{g/L}$ intermediate calibration standard, prepare the following working standards:

Standard Concentration, $\mu\text{g/L}$	100 $\mu\text{g/L}$ Intermediate Cal Standard, mL	Final Volume
2.0	2	100
5.0	5	100

The working calibration standards are digested along with the samples using the procedure outlined in Section 7.3.

For soil/tissue samples, pipet an appropriate volume (as specified above) of the intermediate standards into BOD bottles, add the appropriate volume of reagents and dilute to 100 mL with DI water after digestion.

NOTE: Any standard may be prepared from any stock or intermediate standard or any volume as long as the appropriate factor is applied and the correct v/v ratio is maintained.

7.5 Initial and Continuing Calibration Verification Standards

For water samples, transfer 100 mL of the working Hg ICV/CCV standard into a 300-mL BOD bottle and add the appropriate volumes of reagents. For soil/tissue samples, pipet 2 mL of the intermediate 100 $\mu\text{g/L}$ standard into a BOD bottle, add the appropriate volumes of reagents and dilute to 100 mL after digestion. The ICV/CCV are digested along with the samples using the procedure outlined in Section 7.3. The ICV will be run immediately following instrument calibration. The CCV will be run after every 10 samples and at the end of the analytical run.

NOTE: Standards may be prepared using alternate intermediate standards as long as the final concentration is 2 $\mu\text{g/L}$ and the v/v ratios are maintained.

7.6 Laboratory Control Sample

Laboratory control samples (LCS) are typically either a secondary or tertiary source from a certified vendor. The LCSs are prepared for use as per the vendor's instructions. Alternatively a blank spike may

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be prepared by spiking DI water or sand with a known amount of Hg (at or near midrange of the calibration curve) using the second source Hg standard. The LCS will be analyzed with the frequency of one per batch of 20 samples of the same matrix. Prepare the LCS using the procedures outlined in Section 7.3 at the same time the samples are prepared.

7.7 Method and Initial/Continuing Calibration Blanks

A method blank (MB) using 100 mL of DI water must be prepared for each analytical batch of samples not to exceed 20 samples. This blank is also used for dilution water, the initial calibration blank (ICB), the continuing calibration blank (CCB) and as the “zero” point for the initial calibration. Digest as in Section 7.3.

7.8 Matrix Spike/Matrix Spike Duplicate

Prepare a MS/MSD sample by spiking a sample with a known amount of Hg approximately midrange of the calibration curve at a frequency of one in ten samples or per project. Prepare the MS/MSD for water samples by spiking 2 mL of the 100 µg/L intermediate calibration Hg standard to 100 mL of water for a concentration of 2 µg/L. Prepare the MS/MSD for solid samples by spiking 2 mL of the 100 µg/L intermediate calibration Hg standard to 0.2 to 0.8 g of sample. Digest using the procedures outlined in Section 7.3.

7.9 Calibration and Measurement

Differences between the various makes and models of satisfactory atomic absorption (AA) instruments and cold-vapor generators prevent the formulation of detailed instructions applicable to each system. The analyst should follow the manufacturer's operating instructions for a particular instrument and cold-vapor generator system.

Analyze the full set of working calibration standards using two replicates of each standard at the beginning of the run to establish the initial calibration curve. The average of each standard's response is used to generate the linear curve. The % relative standard deviation (RSD) should be <20% excluding the zero point.

Analyze calibration verification standards (ICV/CCV) and calibration blanks (ICB/CCB) during sample analysis to confirm instrument response at the frequency in Section 9.0. Method blanks, samples and QC samples are analyzed using two replicates during the run and the average value is reported. The % RSD should be <20%.

Samples with concentrations of Hg over the high standard must be diluted into the linear calibration range (see Section 9.8). All sample dilutions will be documented on the “Dilution Preparation and Analysis for Hg” log included in the data package.

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8.0 CALCULATIONS

8.1 Mercury Concentrations

The Hg concentration in $\mu\text{g/L}$ is available directly from the read-out system of the instrument. Using the Leeman automated Hg system, the concentration in the sample is calculated from the instrument's software using a linear fit. The final Hg concentration in the sample is calculated as follows:

$$A = \mu\text{g/L Hg in sample} = B \frac{D + C}{D}$$

where:

B = Concentration of Hg from the calibration ($\mu\text{g/L}$)
C = Amount of acid blank matrix used for dilution (mL)
D = Sample aliquot used for dilution (mL)

Note: If no dilution was required, $C = 0$, and $A = B$.

For solid samples, calculate concentrations as mg/kg based on wet weight:

$$\text{mg/kg Hg in sample (wet basis)} = \frac{B \times V}{W \times 1000} \times DF$$

where:

B = Concentration of Hg from the calibration ($\mu\text{g/L}$)
V = Final volume of the processed sample (mL)
W = Weight of sample (grams)
DF = Dilution factor for diluted samples (1.00 with no sample dilution)

For solid samples, report Hg concentrations as mg/kg based on dry weight:

$$\text{mg/kg Hg in sample (dry basis)} = \frac{\text{mg/kg (wet basis)}}{(S / 100)}$$

where:

S = percent total solids in the sample

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8.2 MS/MSD Samples

MS/MSD percent recovery (%R) must be within 75-125 percent and calculated as:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

where:

SSR = Spiked sample result
SR = Sample result
SA = Spike added

The Relative Percent Difference (RPD) for the MS/MSD samples should be within the advisory limit of ± 20 percent and calculated as:

$$RPD = \frac{|(S - D)|}{(S + D)/2} \times 100$$

where:

S = %R for BS sample result
D = %R for BSD sample result

8.3 LCS Recoveries

The recoveries of each of the compounds in the LCS solution will be calculated using the following equation:

$$\text{Laboratory Control Sample Recovery (\%R)} = (LCSR - B)/SA \times 100$$

where:

LCSR = Concentration of target analyte in LCS
B = Concentration of target analyte in blank
SA = Concentration of spike added or certified value of target analyte

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9.0 QUALITY ASSURANCE/QUALITY CONTROL

All quality control data should be documented and available for reference or inspection at the REAC facility.

9.1 Instrument Calibration

A minimum correlation coefficient (r) of 0.995 must be achieved for the linear calibration to be valid. If r is <0.995 , a new calibration curve must be run.

9.2 Initial Calibration Verification

The calibration curve must be verified by the analysis of an ICV standard (at or near mid-range) from an independent source. The ICV result must be within 10% of the true value for the calibration to be considered valid. If the ICV is outside QC limits, the instrument must be re-calibrated.

9.3 Continuing Calibration Verification

The working standard curve must be verified by analyzing the CCV standard (at or near the mid-range) after every 20 sample injections or approximately every 10 samples. CCV results must be within 20% of the true value, or the previous ten samples must be reanalyzed.

9.4 Initial/Continuing Calibration Blanks

The ICB/CCB results must be less than the reporting limit (RL) for the sequence to continue. Two attempts for the ICB are allowed. If the second attempt fails, refill with fresh ICB/CCB solution and re-calibrate prior to analysis.

9.5 Method Blank

A method blank using the same volume of DI water must be prepared for each analytical batch of samples not to exceed 20 samples. The method blank result must be less than the RL. A method blank containing an analyte concentration $>RL$ may be used in instances when the sample concentrations are at least 10 times the method blank concentration.

9.6 Laboratory Control Sample

The LCS (Section 7.6) must be prepared with each batch of 20 samples and must be within the Performance Acceptance Limits (PALs) supplied by the vendor. If a LCS is not available, a blank spike (BS) may be substituted. The BS level should be at or near mid-range of the calibration, and spike recovery must be within 75-125%. Recoveries are calculated as in Section 8.3. If the LCS is outside QC criteria, the whole batch of samples must be re-digested and re-analyzed.

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9.7 Matrix Spike/Matrix Spike Duplicate

At least one MS and one MSD sample must be digested with every 10 samples, or with each project type to verify the accuracy of the method. The spike level should be at or near mid-range of the calibration. In the event there is not sufficient sample available in the batch to run a MS/MSD, a LCS/laboratory control sample duplicate (LCSD) must be run. Recoveries are calculated as in Sections 8.2 (MS/MSD) or 8.3 (LCS/LCSD).

The Relative Percent Difference (RPD) of MS/MSD or LCS/LCSD samples must be within $\pm 20\%$ and calculated as in Section 8.2.

9.8 Dilution Analysis

If the Hg concentration of any sample exceeds the initial calibration range, the sample must be diluted and reanalyzed. Use the results of the original analysis to determine the approximate dilution factor required to get a Hg concentration within the initial linear calibration range. Dilute the sample and analyze the diluted sample aliquot. Report results from the lowest dilution factor which produces a Hg concentration in the linear calibration range (see Section 8.0).

9.9 System Routine Maintenance

Routine maintenance is conducted using the guidelines provided by the manufacturer as follows:

Maintenance	Frequency
Clean optical cell	As needed
Clean optical windows	As needed
Adjust Hg lamp	Every 3 months or as needed
Replace pump, waste drain and process tubing	As needed
Check pump head	As needed

9.10 Initial Demonstration of Capability

Initial proficiency in Hg analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or for instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicates of a well-mixed reference standard is analyzed using the procedures outlined in this SOP. Calculate the average mean in $\mu\text{g/L}$ and the standard deviation (s) in $\mu\text{g/L}$ for waters and in mg/kg for solid samples.

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The QAO will tabulate the results from all of the analysts per matrix per parameter, and calculate control limits.

9.11 Method Detection Limit Studies

Method detection limit (MDL) studies will be run on an annual basis for the water and soil matrices to verify the minimum concentration that can be measured and reported with 99% confidence. A minimum of seven replicates will be used for the study (EPA 1984).

9.12 System Troubleshooting

If the instrument will not start, check to make sure the power supplies are ON, argon gas is ON and at the correct pressure, exhaust is ON, sample tubing is connected and correct tension on the pump winding tubing. After making these adjustments and the instrument will still not start, power down the instrument, wait a few minutes and then power up. If repeated attempts fail to start the analyzer, contact the manufacturer for assistance.

If high RSDs are encountered during analysis, recheck pump winding tubing, sample uptake probe, stannous chloride line from the solution to the pump, drying tube and points of tubing connection. Adjust and re-calibrate instrument prior to any analysis.

For any hardware/software problems, contact the manufacturer for assistance.

9.13 Nonconformance Memo

A nonconformance memo will be generated any time an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the Quality Assurance Officer for verification of corrective action.

10.0 DATA VALIDATION

Data will be assessed for usability by the Data Validation and Report Writing Group using the most current version of REAC SOP #1017, *Data Validation Procedure for Routine Inorganic Analysis*. However, data is considered satisfactory for submission purposes when all of the requirements listed in the method are met.

11.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for following the chemical hygiene plan and laboratory safety program regarding the safe handling of the chemicals specified in this method.

STANDARD OPERATING PROCEDURES

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DETERMINATION OF MERCURY BY COLD-VAPOR ATOMIC ABSORPTION (CVAA) (EPA/SW-846 Methods 7000A/7470A/7471A)

When working with potentially hazardous materials, refer to EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to REAC SOP #3013, *REAC Laboratory Safety Program*, and REAC SOP #1501, *Hazardous Waste Management*. The analyst should consult all appropriate MSDS information prior to running an analysis for the first time.

12.0 REFERENCES

Leeman Labs. 1997. *AP/PS200II Mercury Analysis Systems Manual*, 150-00128, Revision B.

Leeman Labs. 2000. *AP/PS200II/WinHg Mercury Analysis System*, 150-00202, Revision A.

National Environmental Laboratory Accreditation Committee (NELAC), *Quality Systems*, current approved version.

U.S. EPA, Office of Solid Waste and Emergency Response. 1992. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed. Update I. Method 7000A.

U.S. EPA, Office of Solid Waste and Emergency Response. 1994. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed. Update II. Method 7470A.

U.S. EPA, Office of Solid Waste and Emergency Response. 1994. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed. Update II. Method 7471A.

U.S. EPA. 1984. Federal Register, 40 Code of Federal Regulations (CFR) Part 136, Appendix B, *Definition and Procedure of the Determination of the Method Detection Limit - Revision 1.11*, October 26, 1984.

13.0 APPENDICES

This section is not applicable to this SOP.